# SHEEP AND GOAT'S MILK ANALYSES : CHOICE OF METHODS AND STANDARDISATION

**The aim of this study on sheep and goat's milk analyses is to make an inventory presenting the results of the thought given to the subject by CECALAIT after being asked by the interprofession. It also gives an account of the results from the studies led on an international level (IDF / ISO / AOAC) as they were presented during the IDF Analytical Week last May. It looks at the various analysis methods saying if they can or cannot be used for the analysis of sheep and goat's milks. And finally, it defines the questions that are still to be examined.**

In the normative (AFNOR, IDF / ISO), analytical or legislative literature describing milk analyses methods, the word "milk" means in fact "cow's milk." Consequently, these methods are not applicable as such to milks from other species. That is why CECALAIT, but IDF too, are taking an interest in the choice of methods and in the standardisation of sheep and goat's milk analyses. The criteria usually concerned by milk payment are kept in this inventory : fat, protein, freezing point detection, somatic cells count, microbiological methods and inhibitors detection as well as the chemical reference methods, official methods for milk payment and instrumental methods.

## 1 - GOAT'S MILK

### 1.1 - Determination of fat content in goat's milk

Today, there does not exist a reference method at the international level (IDF / ISO /AOAC) for the determination of fat in goat's milk. The existing methods being standardised for cow's milk only (ISO 1211 : 1999 - IDF 1D:1996 - AOAC 989.05 Milk - Determination of fat content- Gravimetric method (Reference method) (Röse-Gottlieb).

Concerning this criterion, IDF / ISO studies are in progress to determine and characterise a reference method. The method used within this framework is the same as the one used for cow's milk (Röse-Gottlieb's principle).

A collaborative study was carried out to determine fidelity, repeatability and reproducibility parameters, but on seeing the results (Sr=0,021 SR=0,031 in g/ 100g), the IDF / ISO / AOAC / JAT FAT wished it to be renewed (JAT : Joint Action Team).

So a scientific study will be carried out in 2003 ; it will bear more completely on methods and will bring scientific and technical arguments to validate the choice of the method that will be kept. The methods studied will be the Röse-Gottlieb and the etherochlorhydric methods. (Weibull-Berntrop type) (see :ISO 8262-3:1987 Milk products and milk-based foods -- Determination of fat content by the Weibull- Berntrop gravimetric method (Reference method) -- Part 3: Special cases )

The official method for milk payment (Gerber method, NF V 04-210), used today to connect infrared apparatus, is not standardised for goat's milk. Admittedly, its application is possible but it could be marred by mistakes. This is due to the specific characteristics of goat's milk composition (fatty acid composition, free fatty acid content, size of fat globules, seasonal variations…). Moreover, this method is a conventional one that needs to be connected to the reference method.

The instrumental method according to ISO 9622 / IDF 141 (infrared) is applicable to goat's milk. An apparatus evaluation protocol, taking into account this type of milk, has just been adopted by the French Ministry for Agriculture Scientific and Technical Commission.

### 1.2 - Determination of protein content in goat's milk

The Kjeldhal reference method (ISO 8968 / IDF 20) is applicable to goat's milk. Moreover, this milk is within the scope of the method's application. Studies are in progress at the international level to include fidelity values in the normative text.

On the subject, a collaborative study was carried out to determine fidelity parameters, but on seeing the results (Sr=0,015 SR=0,038 in g/ 100g), the IDF / ISO / AOAC / JAT Protein wished it to be renewed.

To this day, the official method for milk payment which applies to goat's milk is the same as the one applied to cow's milk (Amido black routine method following NF V 04-216)

Due to a potential difference in protein composition (caseins, whey proteins) between this two types of milk, it seems advisable to confirm and adjust the validation, if need be, in order to make the connection between the Black amido method on goat's milk to the Kjeldhal reference method.

The instrumental method following ISO 9622 / IDF 141 (infrared) is applicable to goat's milk. An apparatus evaluation protocol, taking into account this type of milk, has just been adopted by the French Ministry for Agriculture Scientific and Technical Commission.

### 1.3 - Freezing point detection in goat's milk

The current reference method (ISO 5764 : IDF 108 - plateau seeking) applied to cow's milk is applicable to goat's milk too.

Studies on the subject are in progress at the international level. They aim to extend the domain of application of the current method to milk from other species.

On the subject, a collaborative study was carried out to determine fidelity parameters, but on seeing the results (Sr=1,5m°C SR=3,1m°C), the IDF / ISO / AOAC / JAT Water wished it to be renewed.

The fix time instrumental method (infrared) used for screening, is applicable to goat's milk. An apparatus evaluation protocol, taking into account this type of milk, has just been

adopted by the French Ministry for Agriculture Scientific and Technical Commission.

**1.4 - Somatic cells count in goat's milk**

The automatic counters are calibrated with standard cow's milk, the reference value of which are determinated by the reference method (IDF 148-1).

This reference method, using visual counting with a microscope, does not include goat's milk in its domain of application. Eventually, it is planned to include milk from other species but, to-date, no study has been engaged on this subject.

On seeing the particularity of goat's milk (small size of cells, …), it appears that the current reference method (dye used) is not suitable for goat's milk. It is necessary to define a new method, specific to this type of milk. This observation was reinforced by assays carried out on the occasion of the European FAIR Program on the matter.

The instrumental method following the ISO standard 9622 / IDF 141 (infrared) is applicable to goat's milk. An apparatus evaluation protocol, taking into account this type of milk, has just been adopted by the French Ministry for Agriculture Scientific and Technical Commission.

**1.5 - Microbiological methods**

Application of these methods to goat's milk are quite possible (most of them are horizontal methods).

**1.6 - Detection of inhibitors**

There is no study being carried out on that point at national and international levels.

**Assessing the work on goat's milk**

The work in progress, or to be carried out, on goat's milk concerns :

● fat matter :
- determination of a reference method (in progress within IDF)
- connection of the butyric method and fidelity assessment (to be carried out in France)

● protein matter :
- confirmation and / or adjustment of the black amido method connection (to be carried out in France)

● freezing point detection :
- fidelity assessment of the method

● somatic cells count :
- definition of a new reference method (to be carried out in France)
- work on calibration of automated methods(to be carried out in France)

**SHEEP'S MILK**

**2.1 - Determination of fat content in sheep's milk**

For the time being, there does not exist a chemical reference method for the determination of fat in sheep's milk, at French or international levels (IDF / ISO /AOAC). The existing methods being standardised for cow's milk only (ISO 1211 : 1999 - IDF 1D:1996 - AOAC 989.05 Milk - Determination of fat content- Gravimetric method (Reference method) (Röse-Gottlieb).

Concerning this criterion, IDF / ISO work is in progress to determine and characterise a reference method. It is based on the method used for cow's milk analysis but the test portion is divided by two.

A collaborative study was carried out to determine fidelity parameters, but on seeing the results (Sr=0,034 SR=0,065 in g/ 100g), the (IDF / ISO / AOAC) JAT FAT wished it to be renewed.

Concerning the official method for milk payment, a specific standard does exist for sheep's milk but it needs to be connected to the reference method. (NF V04-155 acidobutyrometric method).

The instrumental method following ISO 9622 / IDF 141 (infrared) is directly applicable to sheep's milk. An apparatus evaluation protocol, taking into account this type of milk, has just been adopted by the French Ministry for Agriculture Scientific and Technical Commission.

**2.2 - Determination of protein content in sheep's milk**

The chemical reference method (ISO 8968 / IDF 20 - Kjeldhal) is applicable to sheep's milk.

On the subject, a collaborative study was carried out to determine fidelity parameters, but on seeing the results (Sr=0,036 SR=0,052 in g/ 100g), the (IDF / ISO / AOAC) JAT Protein wished it to be renewed.

Today, the official method for milk payment applied to sheep's milk is the same as the one applied to cow's milk (Amido black routine method following NF V 04-216)

The instrumental method following ISO 9622 / IDF 141 (infrared) is applicable to sheep's milk. An apparatus evaluation protocol, taking into account this type of milk, has just been adopted by the French Ministry for Agriculture Scientific and Technical Commission.

**2.3 - Freezing point detection in sheep's milk**

The current reference method (ISO 5764 : IDF 108 - plateau seeking) applied to cow's milk is applicable to sheep's milk too.

On the subject, a collaborative study was carried out to determine fidelity parameters, but on seeing the results (Sr=1,7m°C SR=4,2m°C), the (IDF / ISO / AOAC) JAT Water wished it to be renewed for confirmation.

The fix time instrumental method (infrared) used for screening, is applicable to sheep's milk. An apparatus evaluation protocol, taking into account this type of milk, has just been

adopted by the French Ministry for Agriculture Scientific and Technical Commission.

**2.4 - Somatic cells count in sheep's milk**

The automatic meters are calibrated with standard cow's milk, the reference value of which is determinated by the visual count reference method (IDF 148-1).

This reference method is directly applicable to sheep's milk, as attested by assays carried out on the occasion of the FAIR European program on that point. But, fidelity parameters are still to be determined.

The instrumental method following the IDF 1418-3 is directly applicable to sheep's milk, but it still needs an adaptation of the operating process to the high fat and protein contents. An apparatus evaluation protocol, taking into account this type of milk, has just been adopted by the French Ministry for Agriculture Scientific and Technical Commission.

**2.5 - Microbiological methods**

Application of these methods to sheep's milk are quite possible (most of them are horizontal methods).

**2.6 - Detection of inhibitors**

There is no study being carried out on that point at the national and international levels.

**Assessing the work on sheep's milk**

The work in progress, or to be carried out, on sheep's milk concerns :

● fat matter :
- determination of a reference method (in progress within IDF)
- connection of the butyric method and fidelity assessment (to be carried out in France)

● protein matter :
- confirmation and / or adjustment of the black amido method connection (to be carried out in France)

● freezing point detection :
- fidelity assessment of the method

● somatic cells count :
- definition of a new reference method (to be carried out in France)
- work on instrumental apparatus calibrating (to be carried out in France)

Philippe TROSSAT

Abbreviations

FAIR 1 CT 95-0881: European Union program named "Control strategies in farms of sheep and goat's milk somatic cells count "
JAT Joint Action Team
r : maximal difference between duplicates
R : reproducibility limit
Sr : standard deviation of repeatability
SR : standard deviation of reproducibility

Bibliography

- TROSSAT Ph., **L'analyse du lait de chèvre et de brebis**, PréAO sous Powerpoint, AG CECALAIT 27 mai 2003, 28 p.

- TROSSAT Ph., **Note sur l'analyse du lait de chèvre**, CECALAIT, 7 avril 2003, 7 p.